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*Structural and High-Temperature Tensile
Properties of Boron Pyrolytic Graphite*

[W. V. Kotlensky and
H. E. Martens]

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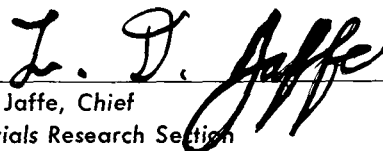
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*Structural and High-Temperature Tensile
Properties of Boron Pyrolytic Graphite*

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ABSTRACT

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Several producers of boron pyrolytic graphite have reported it to have improved mechanical properties over pyrolytic graphite. The tensile properties in the basal plane direction and the structural changes accompanying heating and straining over the range room temperature to 2200°C were found to be similar to pyrolytic graphite. At 2480 and 2760°C pyrolytic graphite with approximately 1% boron exhibits greater ductility than pyrolytic graphite. In comparing the structural transformation of these two materials, the presence of boron appears to enhance the strain induced transformation at 2480 and 2760°C and to inhibit the thermally induced transformation due to heating alone at all temperatures up to 3000°C.

H JTHOR

I. INTRODUCTION

Several producers of pyrolytic graphite (PG) have suggested that the mechanical properties of this material may be improved by alloying it with low atomic weight elements. One alloy which has recently received considerable attention is called boron pyrolytic graphite (BPG) and contains approximately 1% boron by weight in pyrolytic graphite. This material is produced by codepositing from a gas phase boron and carbon on a heated graphite substrate in a manner similar to that used to produce PG (Ref. 1).

The boron atoms in BPG are believed to enter the graphite lattice substitutionally (Ref. 2). In a material

containing 1% boron, uniformly distributed, this means that one boron atom has been substituted for a carbon atom in every 44 unit cells, or there is one boron atom in approximately every 11 Å cube volume element. It has been reported (Ref. 1) that the properties of BPG as determined by chemical analysis, x-ray diffraction, electrical characteristics, and thermal expansion, are distinct from those of PG.

This report presents preliminary x-ray, structural and tensile data on one lot of BPG. Similarities and differences in these data with PG data are noted.

II. MATERIAL TESTED

The BPG used in the present work was produced by the Raytheon Company. It was deposited at 2050°C, had a density of 2.215 g/cm³, and had a reported boron content of 1.25 ± 0.25%. Analysis for boron at this laboratory using a wet chemical method (Ref. 3) gave

a value of 0.98%. In a new material, such as BPG, variations in boron content are likely to exist within a single piece and from block to block even though produced during the same run. Such variations are likely to cause variations in the properties measured.

III. EXPERIMENTAL PROCEDURE

Tensile tests were made using the same equipment used to study the tensile properties of pitch-coke graphites (Ref. 4 and 5) and PG (Ref. 6). The test specimen had a 0.10 in. thick by 0.06 in. wide by 0.75 in. long gauge section with 3/4 in. radii fillets.

The shape of the test specimen affected the accuracy of the recorded data. The total error in stress measurement is estimated to be ±200 psi. The error in strain measurement, which is due principally to the deformation in the filleted regions at the ends of the gauge section, increases with increasing strain. To get an indication of the magnitude of the error caused by this deformation, a series of tests were made using specimens on which grid lines were scribed. By carefully measuring the spacings between grid lines before and after testing to various elongations a correlation between the recorded elongation and the measured gauge section elongation was obtained. In this report the recorded elongation values have been corrected to reduce this source of error, and, therefore, the total absolute remaining error is estimated to be approximately ±1/2% for strains less than 4% and approximately ±3% for strains greater than 15%.

X-ray diffraction patterns were obtained using a 14 cm. diameter Debye-Scherrer powder camera with nickel filtered copper radiation. Unit-cell heights were calculated and averaged from the graphite (002) and (004) lines. Unit-cell widths were calculated and averaged from the (10) and (11) bands for nongraphitized specimens and from the (100) and (110) lines for graphitized specimens. The degree of graphitization was calculated from the unit-cell height and the data from the combined curves of Bacon (Ref. 7) and Franklin (Ref. 8).

Preferred orientation measurements were made on thin rod specimens approximately 0.03 inch square by 1/4 to 1/2 inch long. The specimens were cut with the 1/4 to 1/2 inch dimension parallel to the substrate direction. For the specimens cut from the gauge section of tensile specimens, the 1/4 to 1/2 inch dimension was also parallel to the stress direction. The experimental procedure used for making the orientation measurements was the same as that described in Ref. 9 and was similar to that used by others (Ref. 10, 11, 12).

IV. TENSILE PROPERTIES

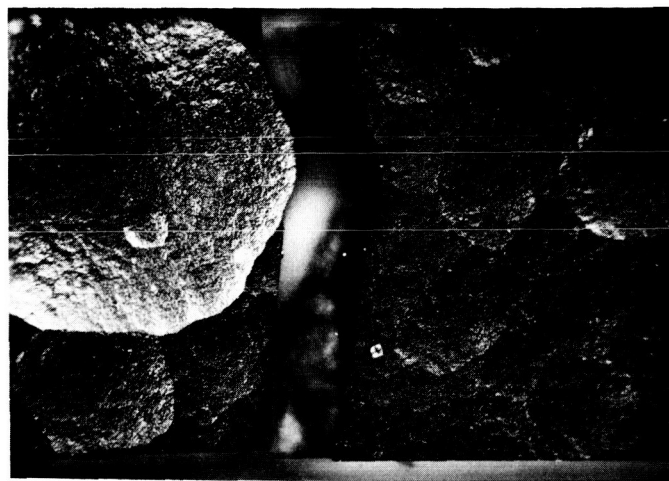
Tensile data for one lot of BPG tested over the range room temperature to 2760°C are given in Table 1. The strength and elongation values for tests 3273, 3274, 3276, and 3278 may be low because fracture occurred at the boundary of a large growth cone, as shown in Fig. 1. Scatter in the strength and elongation values may be due to the presence of large growth cones in the gauge section. If one such growth cone occupies a large percentage of the gauge section, fracture usually occurs at the boundary and low values of strength and elongation are measured. Such a fracture is shown in Fig. 1(a) which is for specimen 3273. Figure 1(b) shows a similar fracture at the boundary of a smaller (0.03 in. dia.) growth cone, which was present in specimen 3274. Specimens 3275 and 3277, which had the highest room temperature strengths, did not have large growth cones in the gauge section. Because of the limited amount of material available, it was not possible to eliminate all large growth-cone areas.

The tensile behavior of BPG can best be described by comparing it with the tensile behavior of PG. Such a comparison is given in Fig. 2 and 3. As seen from the limited data available (Fig. 2), there does not appear to be a marked difference between the tensile strengths

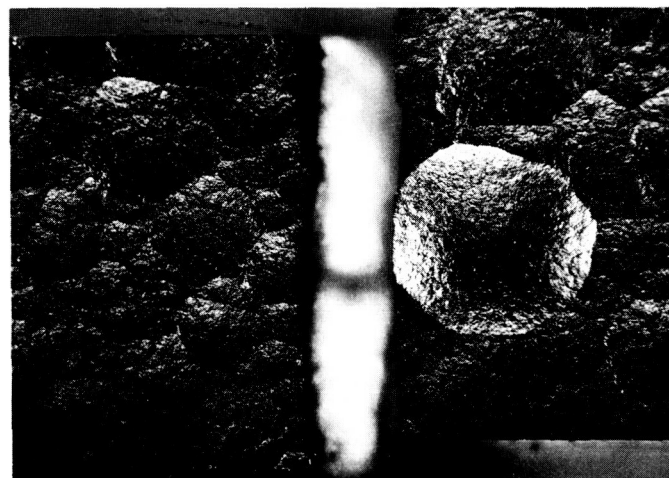
Table 1. Tension test data for lot 189 BPG

Test No.	Test temp., °C	Ultimate tensile strength, psi	Elongation in 0.75 in., %
3273	RT	7,700	0.5
3274	RT	14,400	.9
3275	RT	19,900	1.3
3276	RT	11,900	.6
3277	RT	20,700	.9
3278	1650	15,100	1.6
3279	1650	23,900	3.3
3280	1650	19,700	2.5
3281	1650	18,600	2.5
3282	2200	18,800	3.2
3283	2200	22,700	4.5
3284	2200	30,900	8.5
3285	2200	23,000	5.4
3286	2200	19,800	4.3
3287	2480	46,800	107*
3288	2480	39,100	110*
3291	2760	21,300**	120*
3292	2760	19,900**	116*

Strain Rate 2×10^{-4} sec. $^{-1}$
 * Strain recorder went off scale after approximately 45% recorded elongation. Values given are calculated from direct measurement on specimen after testing.
 ** Specimen did not fracture. Maximum stress reported.



(a)



(b)

Fig. 1. Typical fracture at growth-cone boundary.
Specimen width along fracture
~ 0.060 in. lot 189 BPG

of these two materials over the range room temperature to 2200°C. It should be mentioned that low strengths for PG have also been obtained which were due to fracture at the boundary of large growth cones. The fracture elongation (Fig. 3) appears to be greater for the boron alloy at temperatures of 2200 and 2480°C. This elongation can be clearly seen in Fig. 4 where typical engineering stress-strain curves are shown. Limitations in the test equipment prevented the tests at 2760°C from being carried to fracture.

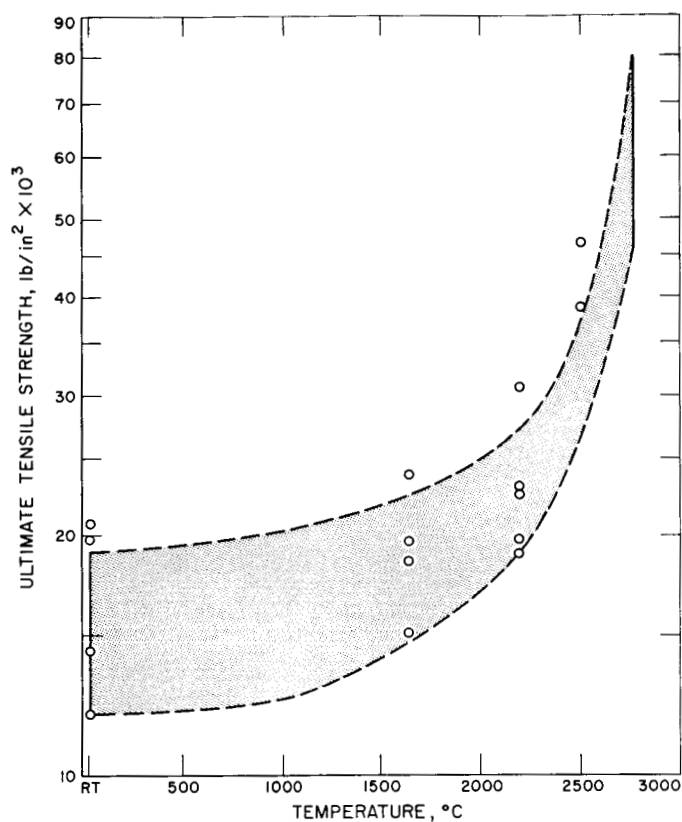


Fig. 2. Ultimate tensile strength vs temperature. Shaded area is for PG. Points are lot 189 BPG

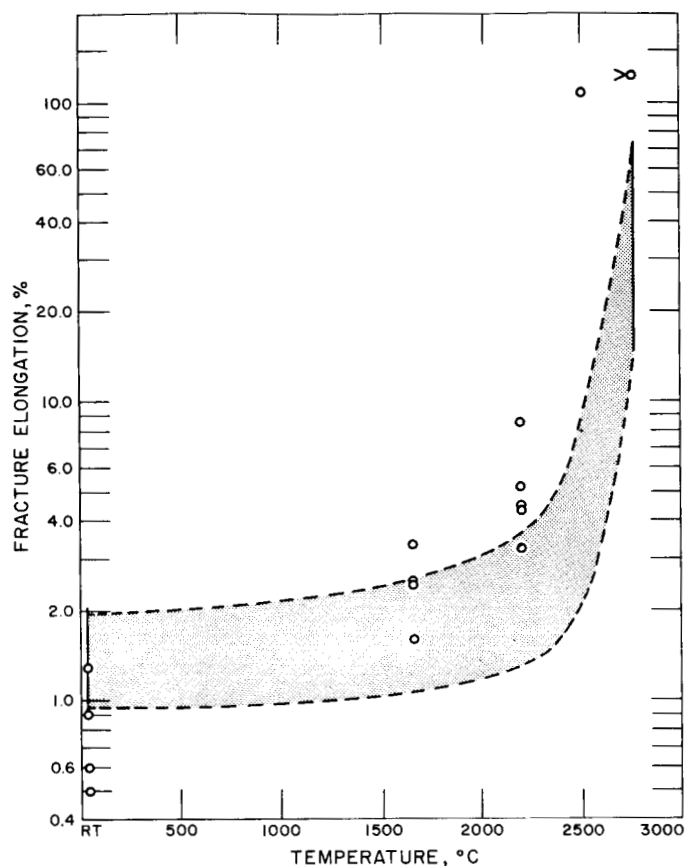


Fig. 3. Fracture elongation vs temperature. Shaded area is for PG. Points are lot 189 BPG

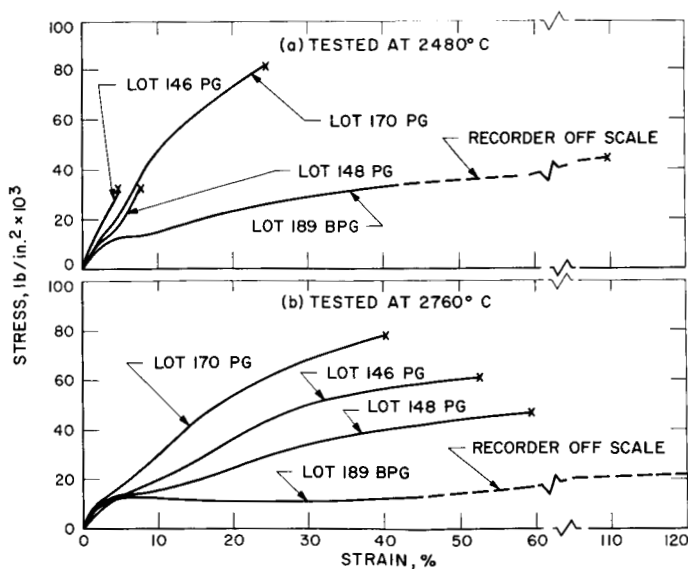


Fig. 4. Typical engineering stress-strain curves for PG and BPG

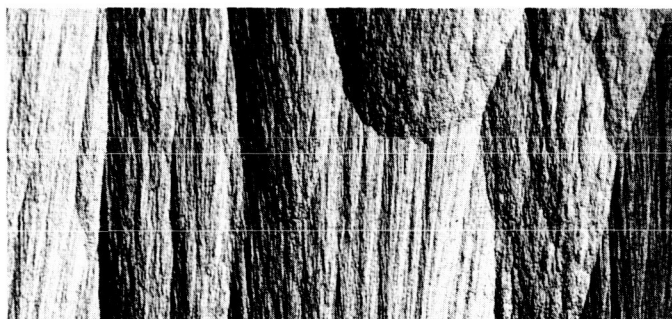
V. STRUCTURAL PROPERTIES

Unit-cell dimensions and percent graphitization for this lot of BPG after heating alone and after heating and straining are given in Table 2. Heating to 2400°C for 30 minutes produced a very slight change in the unit-cell dimensions and degree of graphitization. Heating to 3000°C resulted in an additional small change in these parameters; the change was not as large as might be expected from results for PG (Ref. 6). After heating and straining at 1650 and 2200°C no significant changes were observed in these parameters. The large deformations which occurred at 2480 and 2760°C increased the degree of graphitization to more than 90% and lowered the unit-cell height to approximately 6.708 Å, which is the value reported by Bacon (Ref. 13) for well-crystallized graphites. The unit-cell widths after heating alone and after heating and straining at all temperatures were approximately 2.46 Å, which was approximately equal to the value determined for the as-deposited material.

Table 2. Structural properties for lot 189 BPG after heating and straining.

Temp., °C	Percent strain	Unit-Cell Dimension, Å		Degree of graphitization, %
		c	a	
After only heating				
as-deposited	—	6.793±0.006	2.459±0.003	29
2400	—	6.779±0.02	2.458±0.002	35
2550	—	6.779±0.008	2.458±0.003	35
2700	—	6.760±0.012	2.459±0.004	45
3000	—	6.758±0.008	2.462±0.005	47
After heating and straining				
RT	0.9	6.788±0.02	2.460±0.001	31
1650	2.5	6.782±0.012	2.458±0.003	34
2200	4.3	6.781±0.02	2.458±0.003	34
2480	~107	6.712±0.004	2.462±0.001	92
2760	~120	6.709±0.004	2.464±0.001	98

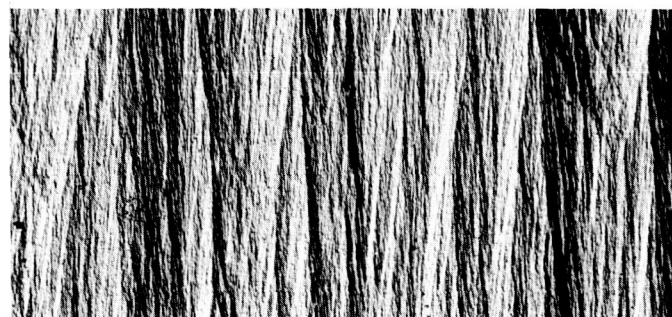
Photomicrographs of the edges of the basal planes (Fig. 5 and 6) show that marked changes in the growth-cone structure are apparent after large deformations at 2480 and 2760°C. Photomicrographs of the specimens after heating only at temperatures up to 3000°C [Fig. 5(c)] and after straining at 2200°C [Fig. 6(a)] show no significant changes. Some variation in growth-cone structure due to manufacturing conditions has been observed; both regenerative and substrate growth-cone nucleation are present, as seen in Fig. 5.



(a)



(b)

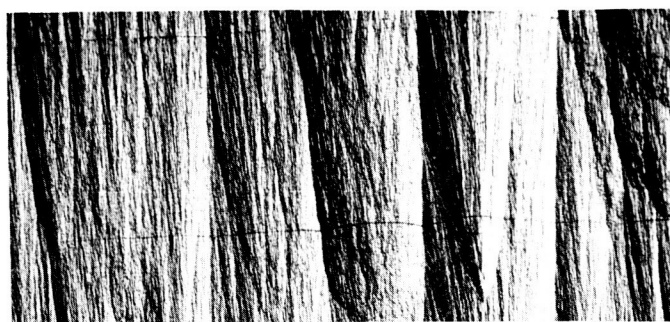


(c)

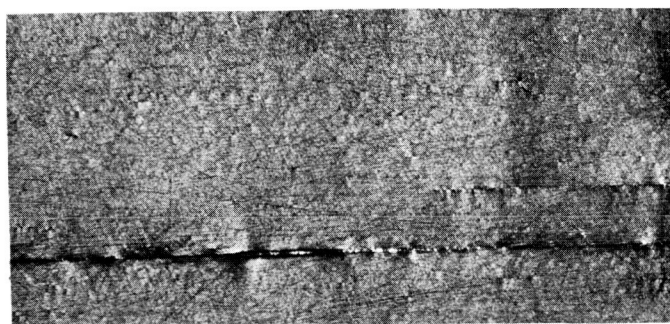
100 μ

Fig. 5. Microstructure of the edges of the basal planes of BPG lot 189 after heating

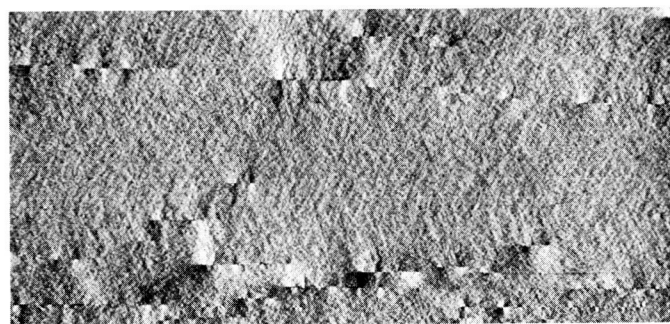
Typical preferred orientation curves after heating and after heating and straining are shown in Fig. 7. These curves are plots of angle of specimen rotation vs the



(a)



(b)



(c)

—100 μ

Fig. 6. Microstructures of the edges of the basal planes of BPG lot 189 after heating and deforming

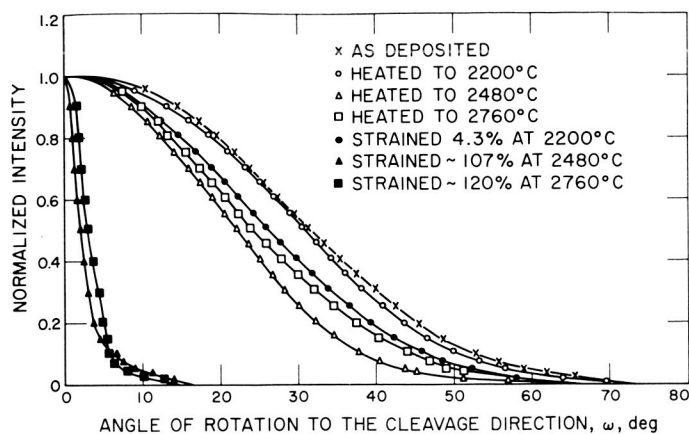


Fig. 7. Typical preferred orientation curves for BPG lot 189

normalized intensity of the (002) peak. In such plots the steeper the curve the higher the preferred orientation. Heating to 2480 and 2760°C causes a greater increase in preferred orientation than straining 4.3% at 2200°C. Following large deformations at 2480 and 2760°C the preferred orientation is greatly increased so that the basal planes are almost perfectly aligned with respect to the stress direction.

Another method, used by others (Ref. 14), to present these changes in preferred orientation is shown in Table 3. Distribution of basal planes as percent of total are tabulated for various angular ranges of inclination to the cleavage direction. Briefly, this distribution was obtained as follows: The area under the preferred orientation curves (Fig. 7) for various inclination angular ranges was assumed to represent the fraction of basal planes inclined to the cleavage direction over that angular range. No corrections were made for instrumental broadening, and, thus, the data presented should be considered on the basis of relative changes. As shown in Table 3, after large deformations at 2480 and 2760°C, approximately 80 to 85% of the basal planes are inclined within 4 deg. to the cleavage direction. After heating to 2760°C and after straining at room temperature and at 2200°C, the distribution of basal planes is not markedly different from the as-deposited distribution.

Table 3. Percent of crystallites inclined at various angles to cleavage direction for lot 189 BPG

Inclination angular range to cleavage direction, deg.	Percent of crystallites inclined at angular range indicated							
	After heating only to, °C				After straining at, °C			
	As-deposited	2200	2480	2760	RT	2200	2480	2760
0-2	6.1	6.3	8.7	7.7	6.4	7.3	62.1	51.3
2-4	6.1	6.3	8.6	7.7	6.4	7.2	23.1	27.9
4-6	6.1	6.3	8.3	7.6	6.3	7.1	9.0	11.9
6-8	6.1	6.2	8.1	7.4	6.2	6.9	4.0	4.2
8-10	5.9	6.0	7.7	7.1	6.1	6.7	1.6	2.3
0-5	15.3	15.8	21.5	19.2	16.0	18.2	90.6	86.7
5-10	15.0	15.3	19.9	18.3	15.4	17.1	9.2	10.9
10-15	14.2	14.4	17.3	16.1	14.2	15.6	0.2	2.4
15-20	12.9	13.1	14.1	13.4	13.1	13.4		
20-25	11.2	11.5	10.5	10.5	11.4	11.3		
25-30	9.2	9.5	7.1	8.2	9.1	8.5		
30-35	7.2	7.2	4.4	5.8	6.7	5.8		
35-40	5.4	5.2	2.9	3.9	4.8	3.9		
40-50	6.2	5.5	1.9	4.0	5.5	4.3		
50-60	2.4	2.0	0.4	0.6	2.5	1.5		
60-70	1.0	0.5			1.3	0.5		

VI. DISCUSSION OF RESULTS

In considering the results presented, it should be pointed out that both BPG and PG are still in the early stages of development. Producers are, therefore, often reluctant to give detailed information on the processing procedure used for a given lot of material. It is recognized that unknown variations in processing could have contributed to some of the differences reported in the tensile and structural properties. Also, processing variations could mask some actual differences which exist but were not observed in this study. Despite the lack of detailed processing information, the structure and deformation behavior of BPG, observed during this investigation, will be compared with the structure and deformation behavior of several lots of PG, studied previously, to gain some insight into the major effects caused by the addition of boron.

When PG is heated above the deposition temperature it undergoes a transformation which results in an increase in preferred orientation, a decrease in turbostratic structure, a decrease in the unit-cell height, permanent growth parallel to the basal planes, shrinkage perpendicular to the basal planes, and elimination of the

growth-cone structure (Ref. 6, 9, 10, 11). The completeness of this transformation, or graphitization as it may be called, depends upon the temperature to which the material is heated. The temperature range for this transformation varies from lot to lot, but, generally, transformation is initiated after heating for 30 minutes at 2600°C and is completed after heating for 30 minutes at 3200°C (Ref. 15). If heating is accompanied by deformation in the basal plane direction, then the completeness is greater at a given temperature, because transformation is initiated at a lower temperature.

In reporting the results of a study on transformation, caused by only heating, Stover (Ref. 11) classified PG's as "easy to transform" or "difficult to transform," depending on the degree of completeness of the transformation after heating to 3000°C.

Based on the microstructure (Fig. 5 and 6), the unit-cell dimensions (Table 2), and the expansion (approximately 2%) at room temperature after heating to 3000°C, the BPG studied in this investigation would be

classified as "difficult to transform." Based on the as-deposited preferred orientation, the BPG is classified midway between the "easy" and "difficult to transform" PG's. One explanation (Ref. 11, 16, 17) for the difference between the two classifications of PG assumes that shear between the basal plane layers is unimpeded in the "easy to transform" material, and is impeded in the "difficult to transform" material. The presence of soot particles is thought to impede basal plane shear in PG. Moreover, it would seem reasonable to postulate that the presence of boron could also impede basal plane shear and, thus, contribute to the "difficult to transform" behavior of the BPG.

The stability of the boron when BPG was heated and strained at the temperatures used in this investigation must also be considered. In one lot of material with an as-deposited boron content of 1.12%, after heating in argon to 2600°C for 30 minutes, the content was determined to be 1.16%, and after heating in argon to 3000°C for 30 minutes the boron content had dropped to 0.67%. In another lot of material the boron content had dropped from an as-deposited value of 0.8% to a value of 0.7% after heating in argon to 3000°C for 30 minutes. It would seem reasonable, therefore, to assume that at the higher temperatures (above 2600°C), used in this investigation, some loss of boron may have occurred. The effects of temperature and deformation on boron loss are currently being studied.

As mentioned earlier, if a tensile stress were applied parallel to the basal planes at the same time that the PG was heated above the deposition temperature, the structural transformation would occur at a lower temperature than if it were just heated. In one of the approximately 20 lots that have been investigated to date (Ref. 18) this strain induced transformation has occurred at 2480°C. In all the other lots the transformation did not occur at this temperature but did occur at 2760°C. The "easy" or "difficult" classification does not seem to apply to the strain induced transformation. Both types seem to exhibit the same behavior when stress is applied parallel to the basal planes.

The BPG studied in this investigation underwent a strain induced transformation at approximately 2480°C. The engineering stress-strain behavior of this material was markedly different from that of any of the lots of PG tested previously. This difference can be clearly seen in Fig. 4.

Lot 146 PG was classed as "easy to transform" and lot 148 as "difficult to transform"; however, as can be seen (Fig. 4), their engineering stress-strain behavior is not markedly different. Lot 170 classed as "easy to transform" was the one lot of PG which showed a strain induced transformation at 2480°C. At this temperature the engineering stress-strain curve for lot 170 [Fig. 4(a)] is markedly different from the curves of lots 146 and 148 which are typical of all PG's tested by the authors. Lot 189 BPG studied during this investigation, classed as "difficult to transform," showed an engineering stress-strain behavior at 2480°C that was different from any of the PG's [Fig. 4(a)]. At a stress of approximately 10,000 psi, the BPG showed a tendency to continue to deform plastically with little increase in stress followed by a region in which the stress increased almost linearly, but very slowly as the strain increased. Conversely, for the PG's, there was no such tendency, and the stress increased at a much greater rate with an increase in the strain. It is evident from Fig 4(a) that lot 189 BPG has a much higher fracture elongation at 2480°C than any PG. It can also be seen that the engineering stress at fracture for the boron material is slightly higher than for the majority of the PG's, but it is lower than that determined for PG lot 170, which underwent a strain induced transformation at 2480°C.

The engineering stress-strain behavior of these same lots at 2760°C is shown in Fig. 4(b). Once again BPG is markedly different. It shows an extended region of plastic deformation with little increase in stress. Also, at this temperature the fracture elongation of BPG is much greater than that of PG.

It has been proposed (Ref. 9) that deformations in PG greater than 20% would involve mechanisms such as nonbasal slip, or fracture across basal planes followed by basal-plane slip, or basal-plane slip without significant increase in preferred orientation. Judging from the data of this investigation, it would appear that any mechanism used to account for the deformation above 20% would have to consider the apparent enhancing effect caused by the presence of approximately 1% boron.

It is obvious that much more information will have to be obtained before a clear understanding of the effect of boron on the higher temperature tensile behavior of PG can be reached. Work is continuing in a current study of BPG's containing up to 2% boron.

VII. CONCLUSIONS

The conclusions resulting from this study of one lot of BPG containing approximately 1% boron are presented as follows:

1. The structural transformation and unit-cell dimensional changes, which occurred upon heating to 3000°C, classified the material as "difficult to transform."
2. The strain induced structural transformation occurred at 2480°C. For all but one lot of PG the transformation did not occur at this temperature.
3. At temperatures up to 2200°C, the tensile properties of the boron material were not markedly different from PG.
4. At 2480°C the BPG had an engineering fracture strength slightly higher, and a fracture elongation markedly greater than PG.
5. At 2760°C the BPG had an engineering fracture strength markedly lower and a fracture elongation markedly higher than PG.

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